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# PATENT SPECIFICATION

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## DRAWINGS ATTACHED

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## (54) FIBRILLATED LAMINATED PLASTICS FILMS

(71) We, PHILLIPS PETROLEUM COMPANY, a Corporation organized and existing under the laws of the State of Delaware, United States of America, of Bartlesville, Oklahoma, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to improved synthetic fibers and yarns. In one aspect the invention relates to methods of producing improved synthetic fibers. In another aspect the invention relates to a readily crimpable fiber.

The formation of fibers and yarn-like structures from oriented thermoplastic film is known in the art. One method comprises orienting a film of polymeric material and splitting the material to form fibers by brushing, rolling, twisting, rubbing, impacting or striking the oriented material. In another method a narrow strip of film is simply uniaxially oriented to a high degree to form fibers.

As used in this specification, "fibrillation" shall define any process by which fibers, filaments, or yarns are formed from a sheet, strip, or ribbon of film material; "fibrillated" shall designate any fibrous product formed from a sheet of film or the like by fibrillation.

According to the invention there is provided a fibrillated plastics film laminate comprising three or more layers of plastics film bonded together and fibrillated, at least two of said layers being molecularly oriented and at least two of said layers being composed of plastics material having different properties, the direction of orientation of the molecularly oriented layers being the same. The different properties in the materials can result from a difference in composition or can be the result of a difference in processing of the same composition. For example, the laminate can be formed by bonding together films including a sheet of polypropylene and a sheet of polyethylene to produce a composite fiber or the laminate can be formed by bonding together

films including a sheet of low molecular weight polyethylene and a sheet of high molecular weight polyethylene.

In one embodiment, the laminate comprises film layers, which have been oriented to different degrees prior to lamination, to give the desired difference in properties of film of the same composition or to modify the difference in properties of films of different polymeric composition.

The difference in properties of the materials in the laminate determines the properties in the fiber product. For example, oriented polypropylene is used to form a high tenacity fiber but is difficult to dye. Fibers comprising a fibrillated laminate of a polypropylene core sheet covered on either side with a layer of polyvinyl acetate are of high strength and readily dyeable with conventional acetate dyes. In addition this fiber crimps when heat relaxed because of the difference in shrinkage characteristics of polypropylene and polyvinyl acetate.

Further in accordance with the invention, there is provided a method of manufacturing a fibrillated plastics film laminate which comprises bonding together three or more layers of plastics film, at least two of said layers being composed of plastics material having different properties and at least two of said layers being molecularly oriented, to form a fibrillatable laminate in which the direction of orientation of the oriented layers is the same, and fibrillating the laminate. The laminate can be fibrillated by passing it between oscillating rollers or plates, by passing it through a zone of turbulence produced by a high velocity stream of air, or by other known methods.

The invention also provides a method of manufacturing a fibrillated plastics film which comprises bonding together three or more layers of molecularly orientable plastics film, at least two of said layers being composed of plastics material having different properties, to form a laminate, molecularly orienting the laminate such that the direction of

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orientation of the layers is the same and the laminate is fibrillatable, and fibrillating the laminate. In this method the laminate may be uniaxially oriented to a sufficiently high degree by stretching that fibrillation is achieved without a separate fibrillating step.

Other features of the invention will be apparent to one skilled in the art upon consideration of the following description, drawings, and appended claims.

Figure 1 is a block diagram showing the steps of one method of the invention.

Figure 2 is a block diagram illustrating another method according to the invention.

Figure 3 illustrates a third method of forming fibers of the invention.

Figure 4 illustrates a crimped fiber of the invention.

Referring to the drawings, Figure 1 illustrates a process wherein initially a laminate of polymeric thermoplastic material is formed. The laminates can be prepared in any manner known to the industry, such as by heat sealing, the use of mutual solvents, extrusion coating, gluing, and the like. Of course the bonding of the layers must be sufficiently strong so that delamination does not occur during orientation or fibrillation. The laminate can be made from films of any polymer capable of forming a fibrillatable film. The polymeric film retains orientation upon relaxation after stretching. Some of the many polymers which can be used include: vinylidene chloride polymers; polystyrene; polyethylene; polypropylene; polyamides, such as poly(hexamethyleneadipamide), poly(ethylenesubamide), poly(methylene bis-p-cyclohexylene-adipamide), polycaprolactam; and polyesters, such as poly(ethylene terephthalate). Copolymers can also be used. Polyacrylonitrile and acrylonitrile copolymers having at least 85 percent of acrylonitrile units which appear to have two dimensional crystallinity for the purpose of this application are classified as fibrillatable polymers.

The film of the aforementioned polymers can be made in any conventional manner, such as by extrusion, casting or flattening of a blown tubing. The films can be of any width that can be fed to the processing equipment, and they can be doubled or slit to form ribbons of a narrow width prior to fibrillation if desired. The film or strips of film can be cut into staple length if desired, either prior to or after fibrillation.

The film or film laminates can be uniaxially oriented by any means known in the industry, such as by heating to a temperature below the melting point of the lowest-melting polymer and stretching or by cold drawing. In the case of film extrusion, the drawing step can be combined with the extrusion step. The ratio of the stretched length to the original length is usually at least 3:1, and is usually in the

range of 4:1 to 16:1. However, higher draw ratios can be used if desired.

The uniaxially oriented laminate can be fibrillated in any manner known to the art, such as by the techniques described in U.S. Patent 3,003,304.

Depending upon the characteristics imparted to the fiber, the fibers may be passed on to another processing step such as heat shrinkage or dyeing or they can be packaged for shipment or cut into staple length.

FIGURE 2 illustrates a process wherein a film laminate is slit or cut into strips of desired width. Of course, the laminate may be initially formed from strips having the desired width. Suitable widths for the film strip depend upon the intended use of the fiber. The film strip is then oriented by drawing in its lengthwise direction. Fiber forming tendencies of the film increase with the increased draw ratio and are most evident in drawn highly oriented film strips of a crystalline polymer. The fiber formed by the method of the process shown in FIGURE 2 is comparable to films which have been oriented and then fibrillated as shown in FIGURE 1.

FIGURE 3 illustrates a method of fiber production wherein two films 1, 2 of a laminate are oriented to different degrees prior to lamination. Films 1 and 2 may be of like or different composition. After orientation, which results in or modifies the different properties of the films, lamination is accomplished without substantially changing the orientation. Gluing with an epoxy resin is one convenient method of forming such a laminate. The laminate is then fibrillated by splitting. Fibrillation, as hereinbefore defined, encompasses the methods depicted in FIGURES 1, 2 and 3.

FIGURE 4 is a magnified illustration of a composite fiber of the invention which comprises a three layer laminate which has been fibrillated and then heated in a relaxed state with the result that a fiber crimped in a sinusoidal wave is produced. During heat relaxation the fiber is heated to a temperature below the melting point of the lowest melting material in the substantial absence of tension for a time sufficient to induce shrinkage in at least one of the film layers. In this particular embodiment, layer 10, the core material, has a higher melting point than outer layers 11 and 12 which are composed of like material. Of course, the three layer laminate can be formed from film, each having a different composition to form a tricomponent fiber.

The fiber shown in FIGURE 4 may be crimped in a random sine-wave configuration.

Fibers of the invention may be formed from film laminate which, when heated in its relaxed state, imparts a crimping in the form of a helical coil to the fiber.

Most film-forming processes do not produce a perfectly uniform thickness. It is believed that minute differences in thick-

ness of the film used to form the laminate impart a degree of randomness to the properties obtained in the fiber. This randomness is especially noticeable and beneficial in the crimped fibers of the invention.

The following examples will serve to further illustrate the invention.

#### EXAMPLE I

A three layer laminate having outer 0.45-mil layers of low density polyethylene and an inner 0.1-mil layer of polypropylene, as is illustrated in FIGURE 4, was uniaxially oriented by drawing at a 7:1 ratio and a temperature of 203°F. The oriented laminate was air fibrillated at ambient temperature and heated for 5 to 10 seconds at 302°F. in a relaxed state. The resulting fibers were crimped in a random sinusoidal wave configuration and had good bulking but low extensibility.

#### EXAMPLE II

A three layer laminate having 0.1-mil outer layers of polypropylene and an inner core 0.8-mil layer of polyethylene was cut into a 1/16-inch wide strip. The strip was uniaxially oriented at a 7:1 ratio at a temperature of about 200°F. The resulting fiber was heated for 5—10 seconds at about 300°F. in a relaxed state. During heating, the fiber was observed to crimp into a random sine-wave configuration.

#### EXAMPLE III

A three layer, 1-mil thick laminate having 0.1-mil outer layers of polyvinyl acetate and inner core layer of polypropylene was cut into a 1/16-inch wide strip. The strip was fibrillated by uniaxially orienting at a 10:1 draw ratio at room temperature. The resulting fiber was heated in an oven at 302°F. for 5—10 seconds in a relaxed state. The fiber twisted into a helical configuration of random S and Z twist. Examination by microscope revealed that the surface layers of the fiber were fractured.

#### EXAMPLE IV

A strip of film laminate, as described, in Example III, was oriented by drawing to a draw ratio of 10:1 at 170°F. The fiber was then heated to about 300°F. for 5—10 seconds in its relaxed state. The fiber had the appearance of the fiber obtained in Example III except the surface layers were free of fractures.

Other laminates that can be used are polyvinyl acetate-polypropylene-polyvinyl acetate and polyvinyl chloride-polypropylene-polyvinyl chloride.

#### WHAT WE CLAIM IS:—

1. A fibrillated plastics film laminate comprising three or more layers of plastics film

bonded together and fibrillated, at least two of said layers being molecularly oriented and at least two of said layers being composed of plastics material having different properties, the direction of orientation of the molecularly oriented layers being the same.

2. A laminate according to claim 1, composed of three layers.

3. A laminate according to claim 1 or 2, wherein at least one of the layers is polyethylene, polypropylene or polyvinyl acetate.

4. A laminate according to claim 1 or 2, including a layer of polyethylene and a layer of polypropylene.

5. A laminate according to claim 1 or 2, including a layer of polypropylene and a layer of polyvinyl acetate.

6. A laminate according to claim 1 or 2, including a layer of polypropylene bonded between two layers of polyvinyl acetate.

7. A laminate according to any one of claims 1 to 6, wherein the said different properties are the heat shrinkage characteristics of the two layers.

8. A fiber derived from a fibrillated laminate according to any one of claims 1 to 7.

9. A fiber according to claim 9 which is crimped.

10. A fiber according to claim 8 or 9 composed of only two different plastics material.

11. A method of manufacturing a fibrillated plastics film laminate which comprises bonding together three or more layers of plastics film, at least two of said layers being composed of plastics material having different properties and at least two of said layers being molecularly oriented, to form a fibrillatable laminate in which the direction of orientation of the oriented layers is the same, and fibrillating the laminate.

12. A method of manufacturing a fibrillated plastics film which comprises bonding together three or more layers of molecularly orientable plastics film, at least two of said layers being composed of plastics material having different properties, to form a laminate, molecularly orienting the laminate such that the direction of orientation of the layers is the same and the laminate is fibrillatable, and fibrillating the laminate.

13. A method according to claim 12, wherein in the laminate is uniaxially oriented to a sufficiently high degree by stretching that fibrillation is achieved without a separate fibrillating step.

14. A method according to claim 11, 12 or 13, wherein the said different properties are the heat shrinkage characteristics of the two films and the fibrillated product is heated to induce crimping.

15. A method according to claim 11, wherein the said different properties are the degrees of orientation of the oriented films.

16. A method according to claim 11, substantially as hereinbefore described with

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reference to Figure 3 of the accompanying drawings.

17. A method according to claim 12, substantially as hereinbefore described in Example I or II.

18. A method according to claim 13, substantially as hereinbefore described with reference to Figure 2, of the accompanying drawings or Example III or IV.

10 19. A fibrillated laminate when prepared

by a method according to any one of claims 11 to 18.

20. A fibrillated laminate according to claim 1, substantially as hereinbefore described with reference to any one of the accompanying drawings or Examples. 15

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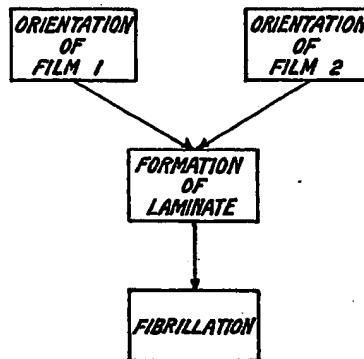
Fig.1.



Fig.2.



Fig.3.



1244860 COMPLETE SPECIFICATION

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Sheet 2

Fig. A

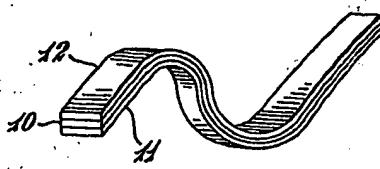


Fig. A shows a strip of metal having a wavy, undulating pattern along its length. The strip is attached to a surface at one end by means of a series of vertical folds or ribs, indicated by reference numeral 10. At the other end, the strip is attached to a surface by means of a series of vertical folds or ribs, indicated by reference numeral 12. The strip is supported by a series of vertical folds or ribs, indicated by reference numeral 11.